

Modelling the role of pore water salinity on the water retention behaviour of compacted active clays

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Summary

Compacted active clays are used as construction material for engineering barriers in many geotechnical and geo-environmental applications. Their very low permeability makes them particularly suitable as liners for the containment of both municipal and radioactive waste, since it allows very limited fluxes of contaminants.

Experimental data show that the water retention behaviour of active clays is very dependent on pore water chemistry, since for a given matric suction the mass of stored water decreases with water salinity. This issue can have relevant effects on the performance of clay liners, considering that active clays are usually cast in place in unsaturated conditions and that the chemical composition of their pore water after compaction may be different from the one of the surrounding environment. In this paper, the effects of water content and water salinity on compacted clay fabric are firstly reviewed, through existing formulations which consider them separately. An enhancement of the existing formulations is then proposed, by merging both contributions. The model is finally validated against experimental water retention data from the literature, showing good prediction capabilities. In the case of the active clays considered, at given water ratios, the model predicts that the matric suction can vary between one and two orders of magnitude with salt concentration. As a counter fact, at a given matric suction and initial dry density, the model predicts that the water retained by samples inundated with distilled water is more than twice with respect to the water retained by samples saturated with a concentrated solution.

Keywords: water retention curve, clay, chemical interaction, suction

1. Introduction

Pore fluid composition has relevant effects on the hydro-mechanical behaviour of compacted clays and its appropriate modelling is essential for geoenvironmental applications such as waste containment barriers and earth constructions. Compacted soils are cast in place in unsaturated conditions with a compaction pore fluid whose chemical composition and species concentration could be significantly different from the one of the surrounding groundwater. Chemical and hydraulic solicitation can be also induced by leachates of municipal and hazardous waste landfills, brine ponds or nuclear waste repositories [THYAGARAJ and RAO, 2014]. In this context, the contribution of both matric and osmotic suction is fundamental to characterize the energy state of pore water. Matric suction is related to capillary and hydration forces acting between the pore fluids and the solid particles: as a consequence, it depends on water content, compaction level and pore size distribution. Compacted clays are characterized by a well-developed structure,

mainly composed of aggregates. Their void space consists partly in intra-aggregate voids (micro-voids) and partly in inter-aggregate voids (macro-voids). For this type of soils, the role of matric suction and water content changes on fabric evolution is well recognized [ROMERO *et al.*, 1999; CUISINIER AND LALUOI, 2004; KOLJI *et al.*, 2006; MONROY *et al.*, 2010]. While fabric changes, the pore size distribution evolves, impacting on the water retention behaviour of the soil [SIMMS and YANFUL, 2004; ROMERO *et al.*, 2011; CASINI *et al.*, 2012; DELLA VECCHIA *et al.*, 2015].

Osmotic suction is viceversa related to the chemical properties of the pore fluid and it depends on the concentration and type of the dissolved species. Although the role of pore fluid chemistry and consequently of osmotic suction on fabric changes has been experimentally detected and modelled for saturated conditions (see, *e.g.* DELLA VECCHIA and MUSSO, 2016), it has received relatively limited attention for unsaturated conditions. However, the usefulness of compacted clays as barrier material strongly depends on the influence of various *chemicals* on their hydraulic response, namely hydraulic conductivity and water retention properties [DUTTA and MISHRA, 2015]. Variations in the chemical composition of the pore fluid induce relevant changes in the fabric of compacted clays, as well documented in literature [MUSSO *et al.*, 2003; MUSSO *et al.*, 2013a; MUSSO *et al.*,

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2013b; CASTELLANOS *et al.*, 2008; SIDDIQUA *et al.*, 2011]. An increase of pore fluid salt concentration causes aggregate shrinkage (reduction of intra-aggregate voids) and, because of the reorganization of the aggregates, also variations of the inter-aggregate pore volume. These variations of macro-pore volume imply relevant changes in the storage capacity of the material, as testified by modifications in the retention properties also in the low suction range.

Coupling between partial saturation effects and chemical composition of the pore fluid has been investigated by [RAO *et al.*, 2006] and by [RAO and THYAGARAJ, 2007], with particular attention to the swelling and compression properties of compacted clays subjected to osmotic gradients. The influence of osmotic suction on the water retention properties of a compacted clay has been firstly tackled by [THYAGARAJ and RAO, 2010], where matric and osmotic suction gradients have been induced by inundating unsaturated clay specimens prepared with distilled water with NaCl solutions or by wetting with distilled water specimens prepared with a salty pore fluid at different concentrations. Experimental results evidenced significant variations of the wetting branch of the water retention curve as a consequence of the difference between reservoir salt solution and pore water, showing the distinct role of matric and osmotic suction dissipation. Evidence of the role of osmotic suction on the water retention properties of compacted Boom clay has been discussed in [MOKNI *et al.*, 2014] on basis of dew-point psychrometer and mercury intrusion porosimeter measurements, showing that, for an imposed water content, varying salt concentration does not only affect total suction – through the osmotic component - but also matric suction. [THYAGARAJ and SALINI, 2015] also found that the chemical composition of the pore water affected both total and matric suction of an expansive clay from Siruseri (India) compacted at different water contents and dry densities. Also in this case matric suction variations have been addressed to modifications in soil microstructure, as testified by ESEM pictures. Similar results have been described for MX80 bentonite specimens in [KUUSELA-LAHTINEN *et al.*, 2016]. The behaviour along drying of GMZ bentonite has been investigated by [HE *et al.*, 2016a; HE *et al.*, 2016b] by means of tests where increasing total suction was imposed, evidencing in this case relevant variations in the water retention properties for specimens prepared at different pore fluid concentrations. Despite emerging laboratory evidences and the need of reliable models to cope with the simulation of the hydro-chemo-mechanical behaviour of compacted clays, no water retention model capable of accounting for the variation of the chemical composition of the pore fluid has been yet proposed. In this paper, a conceptual water retention framework is proposed to reproduce the evolution of hydraulic

properties of compacted clays saturated with fluids whose chemical composition is different from the preparation one. The model stems from the proposal of [DELLA VECCHIA *et al.*, 2015], which relates the evolving retention properties to changes in intra and inter-aggregate void ratio, but it is limited to distilled water as saturating fluid. The original model is here enhanced with a suitable law describing the evolution of the intra-aggregate void ratio with osmotic suction, relying on the microstructural evidences discussed in [MUSSO *et al.*, 2013a].

2. The modelling approach: application to active clays saturated with distilled water

2.1. Constitutive approach and model equations

Water retention curves are usually expressed as a link between matric suction s and a measure of the mass/volume of water in the pores. For deformable porous media, a convenient variable to express the water content is the so-called water ratio e_w , defined as the ratio between the volume of water V_w and the volume of solids V_s . Compacted active clays are generally modelled as double porosity media, the void ratio e being the sum of an intra-aggregate void ratio e_m (equal to the ratio between the volume of intra-aggregate voids and the volume of solids) and an inter-aggregate void ratio e_M (*i.e.* the ratio between the volume of inter-aggregate voids and the volume of solids). Accordingly, the water retention curve proposed in [DELLA VECCHIA *et al.*, 2015] is obtained by splitting the water ratio into two components: the water stored in the aggregates, quantified by the intra-aggregate water ratio e_{wm} , and the water stored in the pores between the aggregates, quantified by the inter-aggregate water ratio e_{wM} :

$$e_w = e_{wm} + e_{wM} = e_m \left[\frac{1}{1 + (\alpha_m s)^{n_m}} \right]^{m_m} + e_M \left[\frac{1}{1 + (\alpha_M s)^{n_M}} \right]^{m_M} \quad (1)$$

e_{wm} is the ratio between the volume of water inside the intra-aggregate pores and the volume of solids, while e_{wM} is the ratio between the volume of water inside the inter-aggregate pores and the volume of solids. α_m , n_m , and m_m are the retention parameters of the micro-structure and α_M , n_M , and m_M are the retention parameters of the macro-structure. It is implicitly assumed that for each structural level a van Genuchten-type water retention relationship holds and that the potential of the pore water within the micro-structure and within the macro-structure is the same. Other possible variables to define the water content are the degree of saturation of the intra- and inter-aggregate voids, $Sr_m = e_{wm}/e_m$ and $Sr_M = e_{wM}/e_M$ respectively.

The air entry value, $1/\alpha$, of the two structural levels is a function of the current fabric of the soil,

Tab. I – Retention parameters for FEBEX bentonite.

Tab. I – Parametri della curva di ritenzione per la bentonite FEBEX.

$\alpha_1^m (MPa)$	$\alpha_2^m (-)$	$n_m (-)$	$m_m (-)$	$a_1^M (MPa)$	$a_2^M (-)$	$n_M (-)$	$m_M (-)$
200	10	7	0.035	10	10	3	0.05

which in the case of active soils evolves along hydro-mechanical paths, as discussed in ROMERO *et al.*, [2011]. The following empirical relationships for the parameters α_m and α_M allow introducing the impact of void ratio changes on the water retention behaviour in a satisfying manner [DELLA VECCHIA *et al.*, 2015]:

$$\frac{1}{\alpha_m} = \alpha_1^m \exp(-\alpha_2^m e_m) \quad (2)$$

$$\frac{1}{\alpha_M} = \alpha_1^M \exp(-\alpha_2^M \frac{e_M}{e})$$

where α_1^m , α_2^m , α_1^M and α_2^M , are model parameters.

Another information that can be obtained by the microstructural analysis of clay samples subject to hydro-mechanical paths is the evolution of aggregate size. According to [ROMERO *et al.*, 2011] and [DELLA VECCHIA *et al.*, 2015], the following expression relates the intra-aggregate void ratio to the water ratio:

$$e_m = \beta_0 e_w^2 + \beta_1 e_w + e_{m,0} \quad (3)$$

where β_0 and β_1 are parameters quantifying the swelling potential of the aggregates and $e_{m,0}$ is the value of the intra-aggregate void ratio for dry conditions ($e_w=0$).

2.2. Model validation

The model described in section 2.1. has been validated in the original paper by simulating water retention data of two compacted medium activity clays, namely Boom Clay and a Sicilian scaly clay, subject to hydro-mechanical solicitations. The first objective of the present paper is extending the proposed model to the case of very active clays, such as bentonites. For this purpose, the retention behaviour of FEBEX Bentonite, which was considered here because of the huge body of experimental data available in the literature, including water retention curves at different densities and under different mechanical constraints. Furthermore, this material has also been characterized in terms of volumetric strains and microstructural changes induced by salinisation and desalinisation processes [CASTELLANOS *et al.*, 2008; MUSSO *et al.*, 2013a].

FEBEX Bentonite is a natural mixed bentonite, which has been proposed as an engineered barrier for radioactive waste disposal [ENRESA, 2000].

Its liquid limit is $w_L = 102\%$, its plastic limit is $w_P = 53\%$ and its clay fraction ($d < 2\mu m$) is 85 %. The first set of data used to validate the model comes from the wetting branches of constant-volume retention curves determined at laboratory temperature by [LLORET and VILLAR, 2007]. Samples were remoulded at the same gravimetric water content ($w = 14\%$) and statically compacted at three different dry densities, namely 1.60, 1.65 and 1.70 g/cm³, corresponding to void ratios of 0.69, 0.64 and 0.59, respectively. The samples were then wetted in isochoric conditions. The experimental relationship between the intra-aggregate void ratio and water ratio is available in ROMERO *et al.*, [2011] and is represented in figure 1 with the predictions based on equation (3). The values of the parameters for FEBEX bentonite follow from this calibration, and they are: $\beta_0 = 0.25$, $\beta_1 = 0.15$ and $e_{m,0} = 0.35$. Water retention parameters for the wetting branch have been calibrated on the $e = 0.64$ curve, according to the procedure described in [DELLA VECCHIA *et al.*, 2015]. The complete set of parameters for the water retention of the FEBEX bentonite is summarized in table I.

The simulations corresponding to $e = 0.69$ and $e = 0.59$ can thus be considered as model predictions. Experimental data and numerical simulations

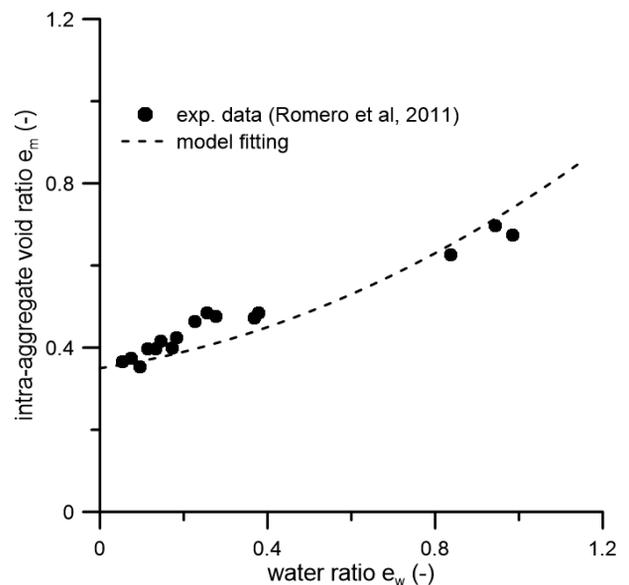


Fig. 1 – Evolution of intra-aggregate void ratio for FEBEX bentonite with distilled water as pore fluid.

Fig. 1 – Evoluzione dell'indice dei vuoti intra-aggregato della bentonite FEBEX esposta ad acqua distillata.

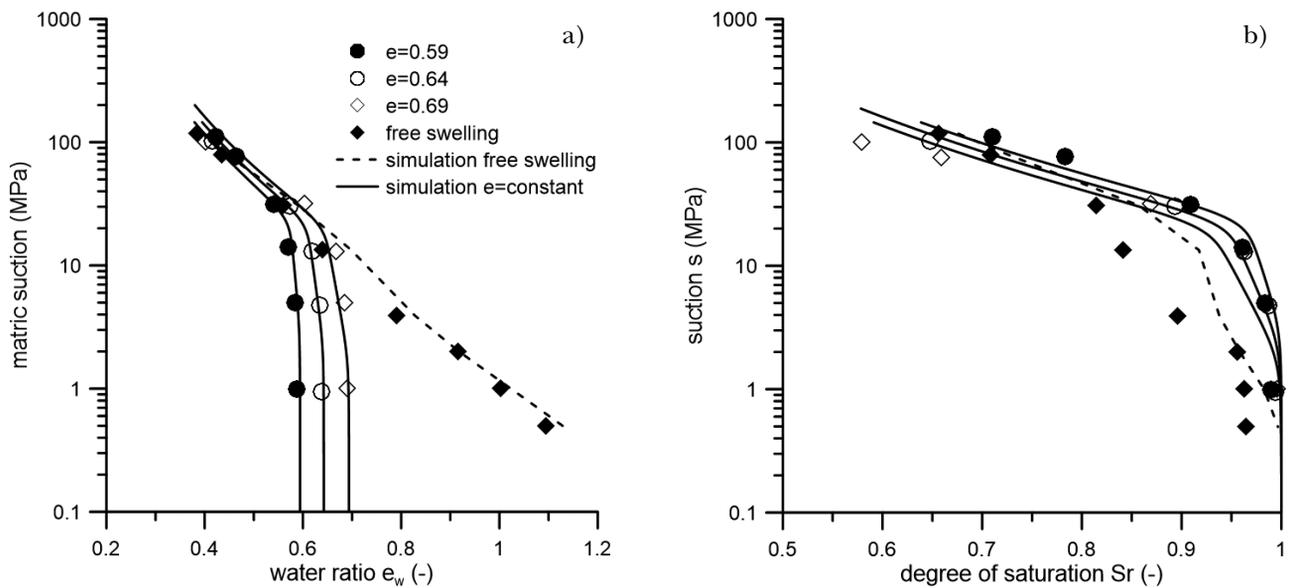


Fig. 2 – Wetting branches of retention curves of FEBEX bentonite prepared at different initial void ratio. a) suction vs water ratio. b) suction vs degree of saturation.

Fig. 2 – Rami di imbibizione di curve di ritenzione di campioni della bentonite FEBEX preparati a diversi indici dei vuoti iniziali. a) in termini di water ratio. b) in termini di grado di saturazione.

(continuous line) for the constant volume retention curves are shown in figures 2a and 2b, in terms of water ratio and degree of saturation evolution with matric suction. Model predictions are satisfactory: the void ratio dependence of the curve is well evident just in the e_w - s plane for low values of suction, i.e. when the aggregates are already saturated and the storage mechanism of water in the inter-aggregate pores dominates. For large suction values, the curves tend to converge, as shown also by the experimental data. Also in the S_r - s plane model predictions and experimental data show a good agreement: the distance between the constant-volume water retention curves in this plane is smaller, but the curves do not coincide because of the void ratio dependency on the air entry value (air occlusion value in this case).

The model calibrated on the constant volume retention data was also used to predict the retention curve of a sample saturated in free swelling conditions. The specimen has been compacted at an initial dry density 1.70 g/cm^3 at the hygroscopic water content ($w = 14\%$). Experimental data in [LLORET and VILLAR, 2007] were complemented with the swelling curves shown in [VILLAR, 2005], providing the evolution of suction with void ratio. Experimental data and model predictions (dashed lines) are also shown in figures 2a and 2b. Also in this case the agreement between experimental data and numerical simulations is pretty good, being the model capable of reproducing both the reduction in the air occlusion value as well as the lower storage capacity: for the same matric suction, the degree of saturation of the swelling sample is low-

er than the one of the sample saturated at constant volume.

An insight on this aspect, which will be crucial to understand the role of pore water chemistry discussed in the next chapter, can be obtained by looking at the model predictions in terms of void ratio and degree of saturation evolution of the intra- and inter-aggregate voids. Under isochoric conditions, e.g. for a fixed $e = 0.64$ (Fig. 3a), the global void ratio remains constant, while the intra-aggregate void ratio evolves as predicted by equation 3. Due to both aggregate swelling upon wetting and the constant volume condition, the inter-aggregate void ratio e_M reduces as long as the water content increases. Vice versa, when the sample is wetted under free-swelling conditions (Fig. 3b), the relative evolution of the total void ratio, estimated from the vertical strain of the specimen, and of the size of the aggregates is such that also the inter-aggregate void ratio is increasing for increasing water content. For a given water ratio, the inter-aggregate void ratio depends then on the type of constraints imposed to the specimen, and it is larger under free swelling conditions than under isochoric conditions. This implies a different degree of saturation evolution in the macro-voids (Fig. 4a), which is responsible for the different matric suctions corresponding to the same water content. In fact, for suctions lower than 10 MPa, the micro-voids are fully saturated (Fig. 4b) and they do not play any direct role on matric suction evolution with water content, while they do indirectly because of invasion of the inter aggregate void space.

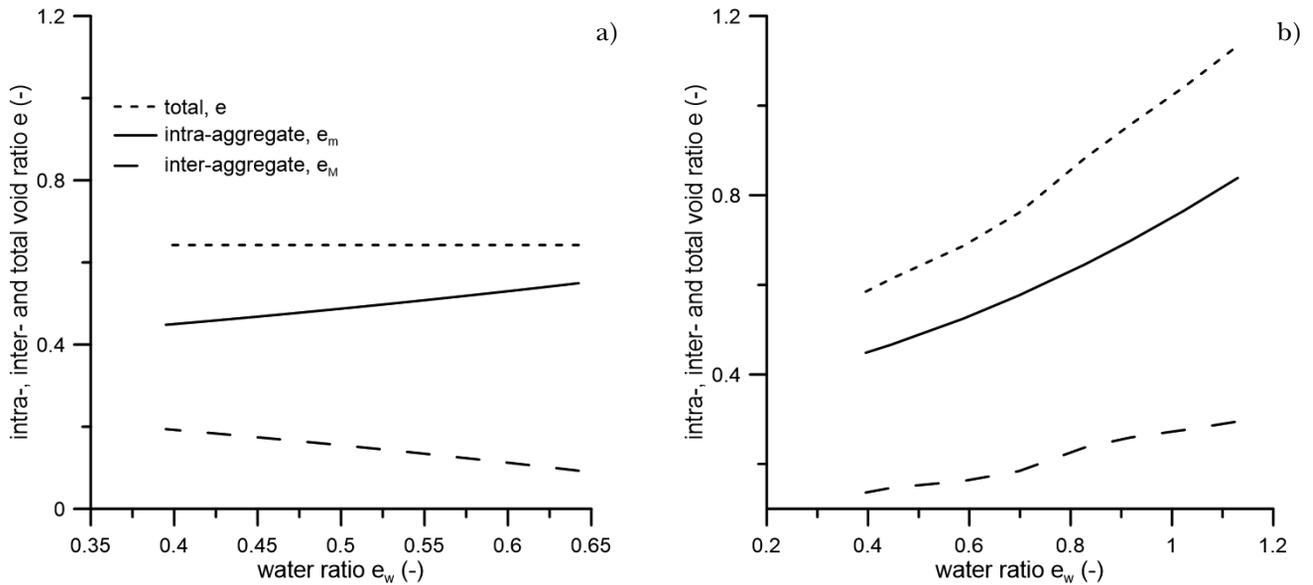


Fig. 3 – Evolution of intra-aggregate, inter-aggregate and total void ratio with water ratio for the FEBEX bentonite. a) constant volume test. b) free swelling test.

Fig. 3 – Evoluzione dell'indice dei vuoti intra-aggregato, inter-aggregato e globale al variare del water ratio nel caso della bentonite FEBEX. a) prova a volume costante. b) prova a rigonfiamento libero.

3. Including the role of pore water chemical composition on water retention on basis of microstructural observations

Active clays are also very sensitive to changes in the composition and concentration of the wetting fluid, as discussed for instance in [BOLT, 1956; MESRI and OLSON, 1971; DI MAIO, 1996]. Repulsion

forces between clay particles in suspension are of electrochemical nature, and depend on the type of cations in solution, their concentration and on the dielectric constant of the wetting fluid. In an unsaturated air-water system, water is the wetting fluid and the dielectric constant does not change. The well-known Gouy Chapman Diffuse Double Layer (DDL) theory neglects the role of the type

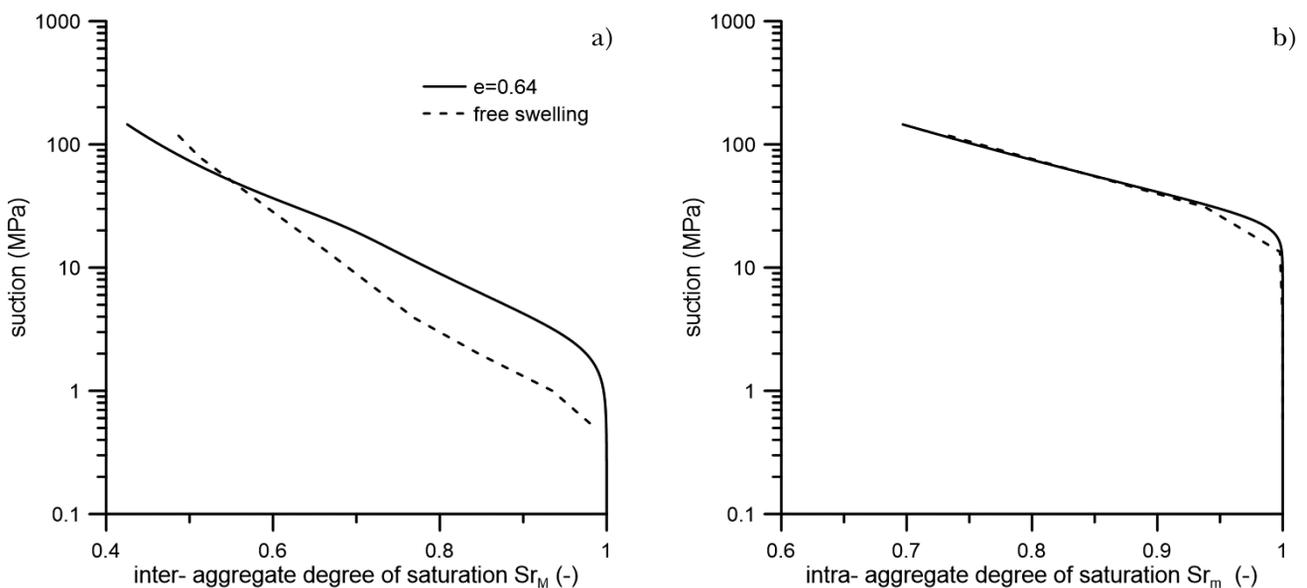


Fig. 4 – Comparison between constant volume and free-swelling tests for the FEBEX bentonite. a) evolution of the inter-aggregate degree of saturation with suction. b) evolution of the intra-aggregate degree of saturation with suction.

Fig. 4 – Confronto tra prova a volume costante e prova a rigonfiamento libero per la bentonite FEBEX. a) evoluzione del grado di saturazione inter-aggregato con la suzione. b) evoluzione del grado di saturazione intra-aggregato con la suzione.

Tab. II – FEBEX bentonite. Experimental intra-aggregate void ratio e_m (from PSDs), prediction for distilled water conditions e_{mw} (obtained from equation 3) and intra-aggregate void ratio reduction due to pore fluid chemistry $e_{mw} - e_m$.

Tab. II – Bentonite FEBEX. Valori sperimentali dell'indice dei vuoti intra-aggregato e_m (da PSD), previsioni per acqua distillata come fluido bagnante e_{mw} (ottenute mediante l'equazione 3) e riduzione dell'indice dei vuoti intra-aggregato dovuta alla composizione chimica della soluzione interstiziale $e_{mw} - e_m$.

Saturating pore fluid	e (-)	e_m (-)	$e_{mw} = \beta_0 e_w^2 + \beta_1 e_w + e_{m,0}$ (-)	$e_{mw} - e_m$ (-)
NaCl 0.5 M	0.878	0.344	0.431	0.087
NaCl 2.0 M	0.770	0.273	0.410	0.137
NaCl 3.5 M	0.750	0.178	0.385	0.207
NaCl 5.5 M	0.740	0.214	0.394	0.180
0.5 M	0.737	0.334	0.428	0.094
2.0 M	0.750	0.145	0.377	0.232
5.5 M	0.750	0.121	0.372	0.251

of cation and explains the interaction between clay particles in terms of concentration of the pore fluid. Accordingly, an increasing concentration causes a reduction of the repulsion forces, reducing the size of the micro-voids. In compacted soils, this ends up with the contraction of the aggregates and therefore with a decrease of the intra-aggregate void ratio.

A convenient expression for the incremental volume strain of the microstructure induced by concentration changes $d\epsilon_{m,vol}^c$, which mimics the predictions of the DDL theory in terms of incremental volume strains, has been proposed by ALONSO *et al.* [1994]

$$d\epsilon_{m,vol}^c = \beta_c \cdot \exp(-\alpha_c \cdot \pi) d\pi \quad (4)$$

where β_c and α_c are two material parameters and π is osmotic suction, which for dilute systems can be expressed by the van't Hoff equation:

$$\pi = i c_s R T \quad (5)$$

being the number of constituents into which the molecule separates upon dissolution (*i.e.* $i=2$ for NaCl solutions and $i=3$ for CaCl₂ solutions), c_s the molar concentration of solute particles, R the universal gas constant and T the absolute temperature in Kelvin.

Introducing the usual assumption of incompressible solid particles, the integration of equation (4) from a reference state where distilled water is the wetting fluid ($\pi = 0$) to a current state where the wetting fluid is a saline solution of osmotic suction $\pi > 0$ provides the following expression for micro-structural void ratio changes due to salinization processes:

$$e_m(\pi=0) - e_m(\pi) = (1+e) \cdot \frac{\beta_c}{\alpha_c} \cdot (1 - \exp(-\alpha_c \cdot \pi)) \quad (6)$$

Then, for a given microscopic water ratio, the corresponding intra-aggregate void ratio will de-

crease as long as pore fluid concentration increases. Postulating that the effects of water content and concentration on the microstructure are independent and additive leads to the following constitutive equation:

$$e_m = e_m(e_w, e_{m0}, \pi) = e_{m0} + \beta_1 e_w + \beta_0 e_w^2 - B \cdot (1 - \exp(-\alpha_c \cdot \pi)) \quad (7)$$

which is an extension of equation 3 to the case of a saline pore fluid. The new constant B has been introduced to make equation (7) more compact.

3.1. Effects of pore water salinity on the intra-aggregate void ratio of FEBEX bentonite in saturated conditions

The influence of pore fluid chemical composition and concentration on clay microstructure was analysed on the base of the conceptual model presented in [MUSO *et al.*, 2013a], that investigated the microstructure evolution of FEBEX bentonite samples subjected to salinization processes starting from the experimental data proposed in [CASTELLANOS *et al.*, 2008]. In order to quantify the effects of salinization on structural evolution, FEBEX bentonite samples at hygroscopic water content equal to 12% were statically compacted to a dry density of 1.65 g/cm³, loaded at constant water content up to 200 kPa and saturated with distilled water, NaCl solutions (0.5 M, 2 M, 3.5 M and 5.5 M) and CaCl₂ solutions (0.5 M, 2 M and 5.5 M). At the end of the wetting stage, the specimens were freeze-dried and subjected to MIP tests to obtain the corresponding pore size distribution.

The values of total, intra- and inter-aggregate void ratios estimated from the experimental PSDs according to the procedure described in [MUSO *et al.*, 2013b] are shown in table II. Since this table refers to saturated conditions, $e_w = e$ holds for all of these samples. The intra-aggregate void ratio predicted on basis of equation (3) appears in the fourth column

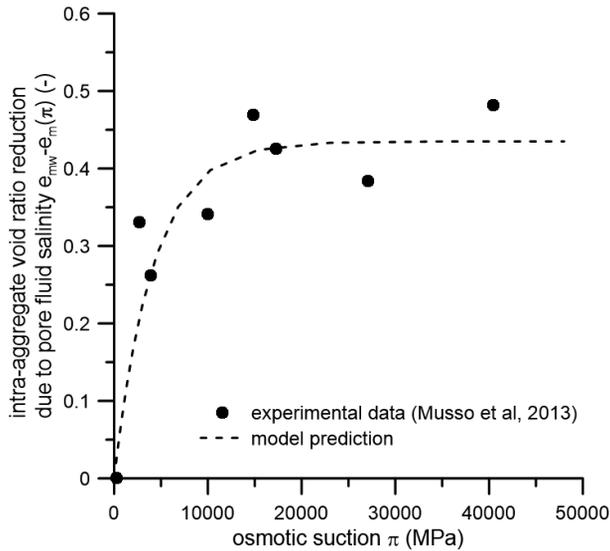


Fig. 5 – Intra-aggregate void ratio variation with osmotic suction for the saturated FEBEX bentonite.

Fig. 5 – Evoluzione dell'indice dei vuoti intra-aggregato dovuta alla suzione osmotica per la bentonite FEBEX in condizioni sature.

as e_{mv} . It can be appreciated that the predicted value is quite larger than the experimental one, and that the difference, provided in the last column of the table, increases with pore fluid salinity. This difference is assumed to be induced by the decrease of the intra-aggregate void ratio caused by the increase in salinity. Equation (7) was thus calibrated on the basis of this microstructural interpretation, obtaining $\alpha_c = 0.26 \text{ MPa}^{-1}$ and $B = 0.43$ and leading to the results shown in figure 5. The overall prediction of the inter-aggregate void ratio for this data set is then given in figure 6, together with the prediction performed with equation (3), *i.e.* for distilled water conditions.

3.2. Modelling the water retention curve

The framework developed for the retention behaviour of double porosity media introduced in Section 2 can be extended to the case of active clays exposed simultaneously to drying/wetting and salinization/desalination processes considering the following main effects:

- Swelling induced by desalination and shrinkage induced by salinization. When salty water is added to an unsaturated clay specimen without any volume constraint, the entity of swelling depends on the chemical concentration of the inflow fluid. This aspect is accounted for by considering different swelling curves in terms of void ratio variation with suction, which depend on the actual chemistry of the pore fluid;

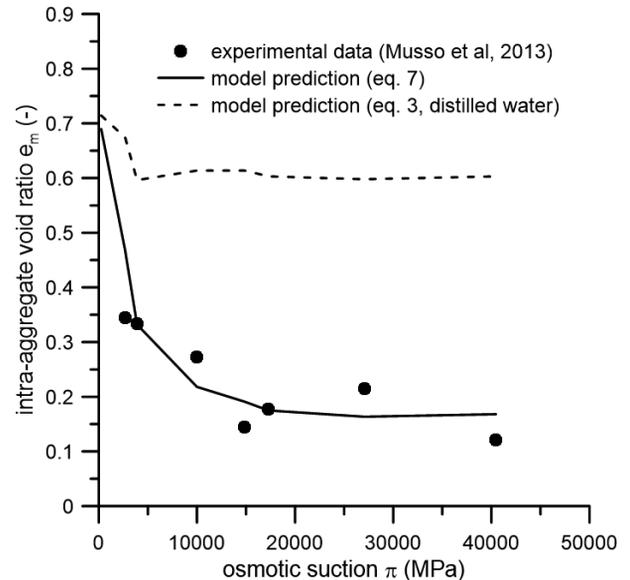


Fig. 6 – Measured and predicted values of intra-aggregate void ratio of FEBEX bentonite in saturated conditions with different saline solutions.

Fig. 6 – Valori misurati e previsioni per l'indice dei vuoti intra-aggregato della bentonite FEBEX saturata da diverse soluzioni saline.

- Dependency of the clay fabric on the chemistry of the wetting fluid. As evidenced for saturated conditions in the previous chapter, the higher the salt concentration of the pore fluid, the smaller the size of the aggregates. The induced fabric changes imply a different distribution of inter- and intra-aggregate pores, causing a variation of the capillary storage effects. This aspect is accounted for by computing the intra-aggregate void ratio with equation (7) instead of equation (3).

The evaluation of the current osmotic suction, *i.e.* of the current molar concentration c of salt in solution within the pore space, requires solving the mass balance equation for the salt species. By adding (or subtracting) a mass of solution having a fixed molar concentration c_s , the current concentration of the interstitial pore water c evolves, and it is different both from its initial reference value c_0 and from c_s . The mass balance of the species in solution can be written as:

$$M_c = M_{c0} + \Delta M_c \quad (8)$$

where M_c is the mass of salt dissolved within the sample at the current configuration, M_{c0} is the mass of salt dissolved within the sample in the initial configuration and ΔM_c is the mass of salt which is added/removed during the wetting/drying process.

The transport processes leading to ion migration (diffusion and advection) and changes in the cationic concentration in the pore fluid are out of the scope of this work. It is instead assumed that, at the scale of

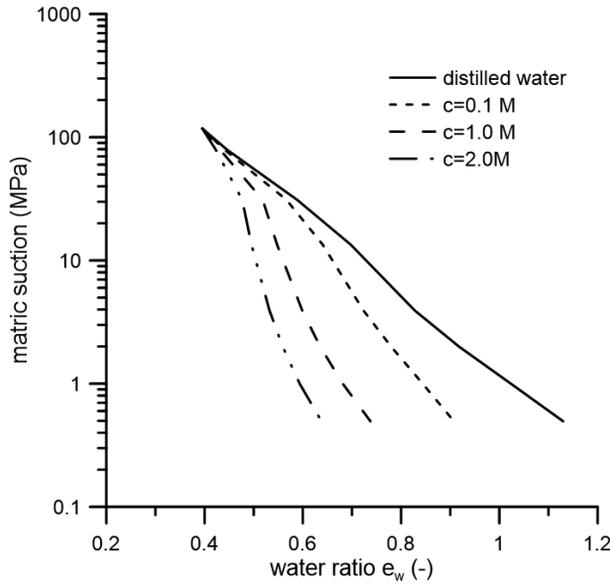


Fig. 7 – Water retention curves for specimens exposed to NaCl solutions: model predictions using FEBEX bentonite parameters.

Fig. 7 – Curve di ritenzione per provini esposti a soluzioni di NaCl: previsioni ottenute con i parametri della bentonite FEBEX.

the laboratory sample, equilibrium has been achieved so that the concentration can be assumed homogeneous.

Under such assumption it follows:

$$\begin{aligned} M_c &= \frac{ce_w}{1+e} V = ce_w V_s \\ M_{c0} &= \frac{c_0 e_{w0}}{1+e_0} V_0 = c_0 e_{w0} V_s \\ \Delta M_c &= c_s \Delta \left(\frac{e_w}{1+e} V \right) = c_s \Delta e_w V_s \end{aligned} \quad (9)$$

where e_{w0} , e_0 , c_0 and V_0 are the initial (reference) values of water ratio, void ratio, concentration and sample volume, respectively. The current concentration c can thus be expressed as a function of the current water ratio and of the concentration of the incoming fluid c_s :

$$c = \frac{1}{e_w} (c_0 e_{w0} + c_s (e_w - e_{w0})) \quad (10)$$

In the particular case of wetting starting from a negligible initial concentration ($c_0=0$), equation (10) further simplifies to:

$$c = c_s \frac{(e_w - e_{w0})}{e_w} \quad (11)$$

Along drying, or when wetting with distilled water, no salt mass is added or removed to the sample, and equation (10) reduces to:

$$c = \frac{c_0 e_{w0}}{e_w} \quad (12)$$

4. Model predictions and comparison with experimental data

4.1. FEBEX bentonite

In order to exploit the capabilities of the model and appreciate the physical consequences of the framework proposed, the model has been firstly applied to FEBEX bentonite, to simulate theoretical wetting paths of water retention curves determined with different composition of the inflow fluid. We assume a material that has been prepared with distilled water as discussed in [LLORET and VILLAR, 2007]: the initial values of suction, water ratio and void ratio have been set equal to $s_0 = 117.7$ MPa, $e_{w0} = 0.394$, $e_0 = 0.585$ and the retention curve for distilled water under free swelling conditions is the one shown in figure 2a. Imagine now to saturate the material with salty water (NaCl) at different concentrations. As a consequence of the wetting process, the specimen will experience some swelling, whose magnitude depends on the chemical concentration of the pore fluid. The model requires the void ratio to be known, and consistent values of void ratios corresponding to every suction have been imposed based on the swelling strains presented in [LLORET and VILLAR, 2007] for specimens saturated with distilled water. Taking the distilled water curve as a reference, swelling paths upon saturation with salty water have been obtained with a simple linear scaling, by imposing a swelling strain at saturation consistent with the experimental data proposed in [CASTELLANOS *et al.*, 2008].

Numerical predictions for matric suction evolution along wetting with water with different NaCl concentrations (0.1 M, 1 M and 2 M) are shown in figure 7. The role of pore water chemistry is evident: the larger the salt concentration, the lower the storage capacity of the clay. In fact, for a given matric suction, the water retained by the sample inundated with distilled water is more than twice with respect to the water retained by the sample saturated with the 2 M solution. This is partly related to the different void ratio evolution and partly related to the different response of the aggregates, which in turn influence the size of macropores. Intra-aggregate void ratio evolution upon wetting, as predicted by the model, is shown in figure 8a as a function of matric suction. The black point represents the as-compacted condition. As expected, the intra-aggregate void ratio increases for the sample saturated with distilled water, according to equation (3). For the specimen saturated with 0.1 M NaCl solution, aggregate swelling is partly inhibited, while for the 1 M specimen the aggregate swelling due to water content change is counterbalanced by aggregate shrinkage due to concentration increase: as a consequence, intra-aggregate void ratio is almost constant upon wetting. Finally, when the specimen is saturated with a 2 M

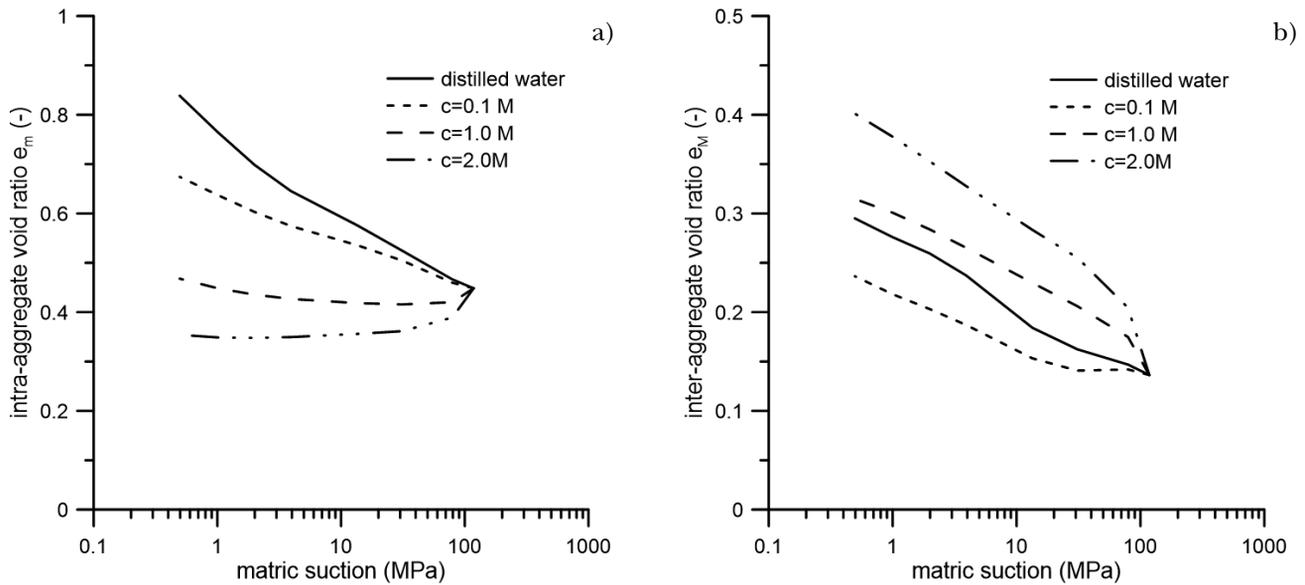


Fig. 8 – Model predictions using FEBEX bentonite parameters for specimens exposed to NaCl saline solutions. a) evolution of the intra-aggregate void ratio. b) evolution of the inter-aggregate void ratio.

Fig. 8 – Previsioni ottenute con i parametri della bentonite FEBEX per l'esposizione di provini a soluzioni saline di NaCl. a) evoluzione dell'indice dei vuoti intra-aggregato. b) evoluzione dell'indice dei vuoti inter-aggregato.

solution, the role of pore fluid chemistry dominates and aggregates reduce their size upon wetting. The combined effect of global and intra-aggregate void ratio evolution upon wetting/salinization influences the volume of inter-aggregate voids: it is evident that inter-aggregate porosity of the 2 M saturated specimen is much larger than the distilled water one. This is the reason for the lower storage capacity in the low matric suction range: larger macro-pores imply a lower degree of saturation for the same matric suction. This aspect is testified also by the predicted evolution of the intra- and inter-aggregate degree of saturation shown in figure 9a and 9b, respectively. Despite no relevant differences are identified in terms of the retention curve of micro-pores, which start to desaturate for suctions larger than 10 MPa, the predicted retention curves for inter-aggregate voids justify the lower storage capacity of specimens subjected to saturation with saline water.

4.2. Karnataka clay

The predictive capabilities of the model have been checked by reproducing the retention curves of another compacted clay, namely Karnakata clay, prepared at a given salt concentration and subjected to wetting with a pore fluid with a different chemical composition, as presented in [THYAGARAJ and RAO, 2010]. Karnakata clay is an expansive clay characterized by a limit liquid $w_L = 82\%$, plastic limit $w_P = 23\%$ and shrinkage limit 10% . The clay was remoulded at a water content $w = 28\%$ by adding

pore fluids with different NaCl concentrations - 0, 0.4 M and 4 M – and then statically compacted in 76 mm-diameter oedometer rings at the optimum Proctor dry density $\rho_d = 1.42 \text{ g/cm}^3$, corresponding to an initial void ratio $e_0 = 0.91$. The specimens (remoulded with distilled water or salty solutions) were then saturated with inflow solutions at different chemical composition. Matric suction during wetting has been determined with triplicate measurements according to ASTM filter paper method, while void ratio changes have been determined via the recorded vertical displacement. During the test, a vertical stress of 6.25 kPa was maintained. The simulation of the whole set of retention curves reported in [THYAGARAJ and RAO, 2010] is here shown. In particular, experimental data involve:

- Test S1: specimen remoulded with distilled water and inundated with distilled water;
- Test S2 and S3: specimens remoulded with distilled water and inundated with NaCl solutions ($c_s = 0.4 \text{ M}$ and $c_s = 4 \text{ M}$, respectively);
- Test S4: specimen remoulded with 0.4 M NaCl solution and inundated with distilled water.

Experimental data concerning the total void ratio evolution with suction, as reported in [THYAGARAJ and RAO, 2010], were used as model input. Water retention parameters corresponding to the part of the model related just to water content variations (*i.e.* equations 1, 2, 3) have been calibrated on the S1 test. In particular, the same values used for Febex bentonite have been adopted to describe the evolution of the intra-aggregate void ratio with water content (Eq. 3). The comparison between model

Tab. III – Retention parameters for Karnataka clay.

Tab. III – Parametri di ritenzione per l'argilla di Karnataka.

α_1^m (MPa)	α_2^m (-)	n_m (-)	m_m (-)	α_1^M (MPa)	α_2^M (-)	n_M (-)	m_M (-)
120	8.9	3.9	0.03	0.09	11	0.7	0.055

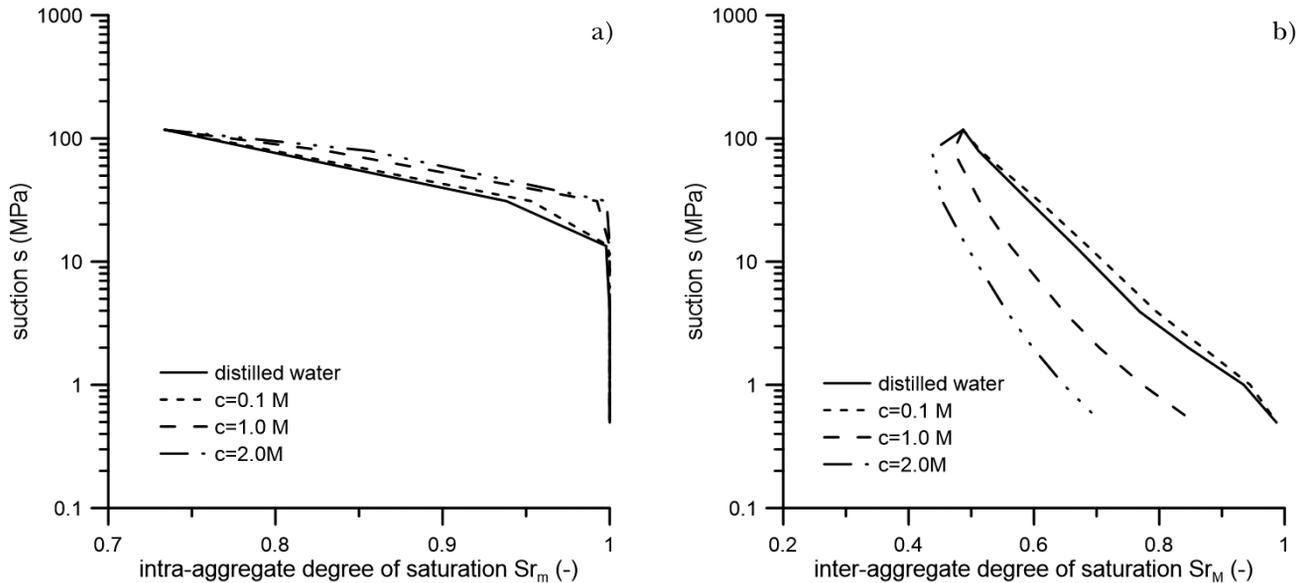


Fig. 9 – Model predictions of water retention curves using FEBEX bentonite parameters. a) water retention for intra-aggregate pores. b) water retention for inter-aggregate pores.

Fig. 9 – Previsioni di curve di ritenzione ottenute con i parametri della bentonite FEBEX. a) curva di ritenzione per i vuoti intra-aggregato. b) curva di ritenzione per i vuoti inter-aggregato.

prediction and experimental data of the S1 test is shown in figure 10. Model parameters are shown in table III.

The role of pore fluid chemical composition has then been accounted for by means of equation (7): the relevant values of $\alpha_c = 11 \text{ MPa}^{-1}$ and $\beta_c = 0.09 \text{ MPa}^{-1}$ have been determined by calibrating model predictions on the experimental data of tests S2 and S3, corresponding to specimens prepared with distilled water and then wetted with different saline solutions (Fig. 10). The current molar concentration was predicted by means of equation (10). The agreement between model predictions and experimental data is remarkably good considering that the role of pore water chemistry is simply accounted for introducing a term which depends on osmotic suction (Eq. 7), which describes the evolution of the inter-aggregate void ratio.

An insight into the simulated evolution of the intra- and inter-aggregate void ratio can help in understanding the role played by salt concentration on model predictions, shown just for tests S1 and S3 for the sake of clarity. Figure 11 shows the major role played by intra-aggregate void ratio evolution on model predictions: while the specimen saturated with distilled water (test S1) experiences an increas-

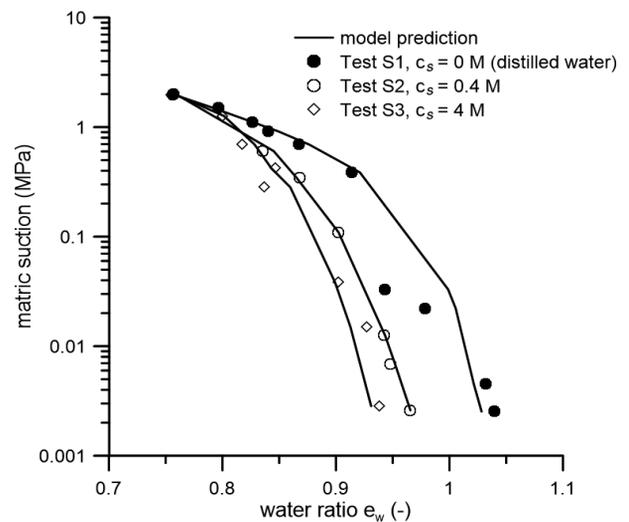

 Fig. 10 – Model prediction and experimental data for specimens of Karnataka clay prepared with distilled water and inundated with NaCl solutions having different concentration c_s . Experimental data in [THYAGARAJ and RAO, 2010].

 Fig. 10 – Previsioni del modello e dati sperimentali per le curve di ritenzione di provini di argilla di Karnataka preparati con acqua distillata ed esposti a soluzioni di NaCl aventi diverse concentrazioni c_s . Dati sperimentali di [THYAGARAJ and RAO, 2010].

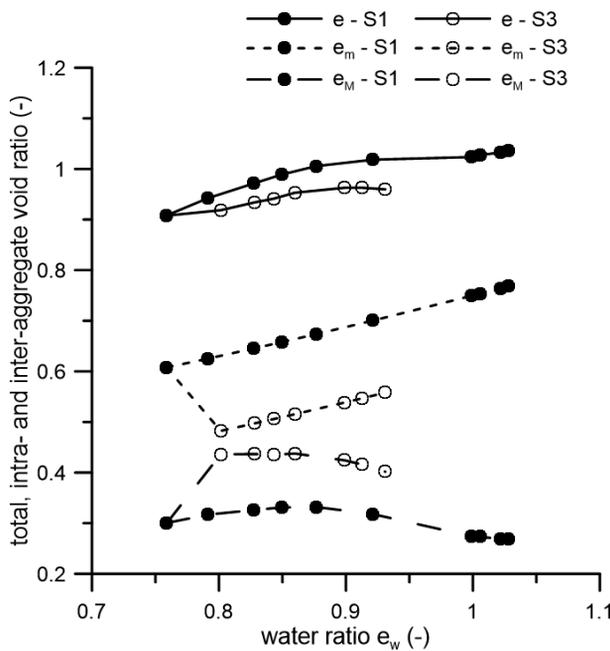


Fig. 11 – Evolution with water ratio of total, intra-aggregate and inter-aggregate void ratio for the simulation of S1 and S3 tests as predicted by the model.

Fig. 11 – Evoluzione con il water ratio dell'indice di vuoti globale, intra-aggregato ed inter-aggregato per la simulazione delle prove S1 e S3 come prevista dal modello.

ing aggregate size for increasing water content, the competing effects of aggregate swelling due to water intake and aggregate shrinkage due to salinization is evident for test S3. The different evolution of aggregate void space with water justifies the different retention behaviour at low suction values: being negligible the differences in terms of void ratio evolution, different aggregate size implies different inter-aggregate void ratios, whose role on water storage capacity is crucial for low matric suction.

The same set of parameters identified for tests S1, S2 and S3 was then used to simulate a free swelling test performed on a salt-amended specimen remoulded with 0.4 M NaCl solution and then inundated with distilled water (test S4 in [THYAGARAJ and RAO, 2010]). The salt mass balance equation to estimate the current chemical concentration of the pore fluid is in this case equation (12). Figure 12 shows the comparison between the predicted retention curve and the experimental data, resulting also in this case in a very satisfactory agreement.

4.3. GMZ bentonite

Other experimental evidences proving the impact of the chemical composition of the pore fluid on the water retention properties have been provided in [HE *et al.*, 2016b] for GMZ bentonite, a Chinese active clay

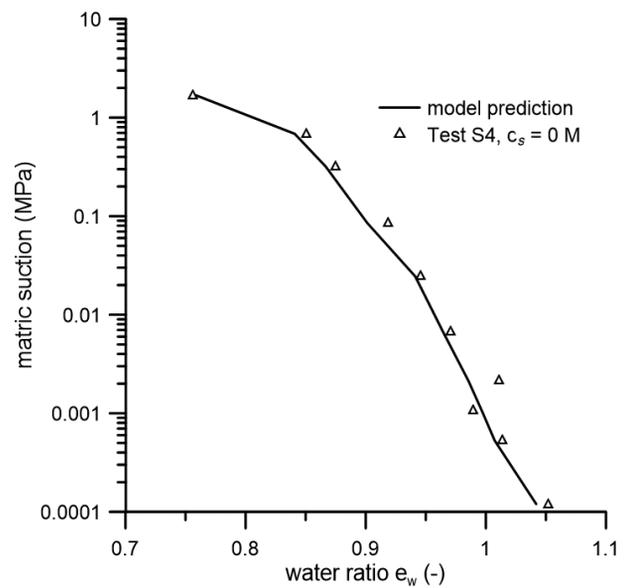


Fig. 12 – Model prediction and experimental data for specimens of Karnataka clay prepared with a $c_0=0.4$ M NaCl solution and inundated with distilled water. Experimental data from [THYAGARAJ and RAO, 2010].

Fig. 12 – Previsioni del modello e dati sperimentali per provini di argilla di Karnataka preparati con una soluzione di NaCl con concentrazione iniziale $c_0=0.4$ M e successivamente esposti ad acqua distillata. Dati sperimentali di [THYAGARAJ and RAO, 2010].

($w_L = 276\%$, $w_P = 37\%$) mainly composed of montmorillonite (75.4%). Compacted specimens of GMZ bentonite have been prepared at compaction water content $w = 10.6\%$ and dry density $\rho_d = 1.7$ g/cm³. Specimens were saturated with distilled water and NaCl solutions at 0.1 M and 1 M concentrations in oedometer conditions under a constant vertical total stress $\sigma = 100$ kPa. As expected, increasing salt concentration of the wetting fluid caused decreasing swelling strains. The saturated specimens, now having different void ratios due to the different swelling, had been then subjected to a drying process, by imposing total suction variations by means of the vapour-transfer technique. Water content changes have been measured too, allowing for a complete characterization of the hydraulic and volumetric state of the specimens at each imposed suction. An attempt to reproduce the experimental data with the water retention model discussed above is here presented. Experimental water ratio and matric suction have been estimated according to the following equations:

$$\begin{aligned} e_w &= S_r \cdot e \\ s &= \psi - \pi \end{aligned} \quad (13)$$

where S_r , e and ψ are the measured/imposed values of degree of saturation, void ratio and total suction, respectively, and π is the osmotic suction calculated by means of the van't Hoff equation. Model param-

Tab. IV – Retention parameters for GMZ bentonite.

Tab. IV – Parametri di ritenzione per la bentonite GMZ.

α_1^m (MPa)	α_2^m (-)	n_m (-)	m_m (-)	α_1^M (MPa)	α_2^M (-)	n_M (-)	m_M (-)
200	9	4.5	0.12	0.5	1.5	0.7	0.22

ters related to distilled water as pore fluid have been calibrated on the basis of the experimental data belonging to the specimen saturated with distilled water and then dried. The parameters related to aggregate swelling due to water content increase have been set equal to $e_{m0} = 0.2$, $\beta_0 = 0.45$ and $\beta_1 = 0.09$, the other material parameters are listed in table IV.

Parameters α_c and β_c related to aggregate size evolution with the chemical composition were obtained exploiting experimental data related to specimen saturated with NaCl saline solution at 0.1 M and 1 M concentration. Since a drying path was imposed, the right expression for the salt mass balance equation is given again by equation (12). Model predictions and numerical simulations are shown in figure 13a and 13b in terms of water ratio and degree of saturation with matric suction. Also in this case the model seems to adequately take into account the relevant changes in clay fabric induced by the variations of the chemical concentration of the pore fluid, which end up affecting the storage properties of the material.

5. Discussion and conclusions

The water retention behaviour of a clay is a function, among other variables, of its fabric. The inter-

action between particles of active clays is substantially ruled by environmental variables such as the water content and the chemical composition of the pore water. Necessarily, the fabric of these soils is by no means a fixed soil property and it is found to vary significantly with both water content and salinity. Modeling the water retention of compacted active clays requires then tracking the effects of environmental actions on fabric.

A common way to account for fabric changes is to adopt a double porosity framework, which identifies a microscopic domain related to the clay aggregates and to the small pores within them, labeled with an intra-aggregate void ratio, and a macroscopic domain, labeled with an inter-aggregate void ratio. Different water retention curves can be defined for each one of the two domains. The overall water retention curve of the material, expressed in terms of water ratio, is the given by the sum of the water retention curves of the two domains. Environmental forces exert measurable effects mainly on the microscopic domain: aggregates swell (*i.e.* the intra-aggregate void ratio increases) for increasing water content and decreasing concentration of dissolved species, while they shrink for opposite processes. On the other hand, the evolution of the inter-aggregate void ratio is very much dependent on

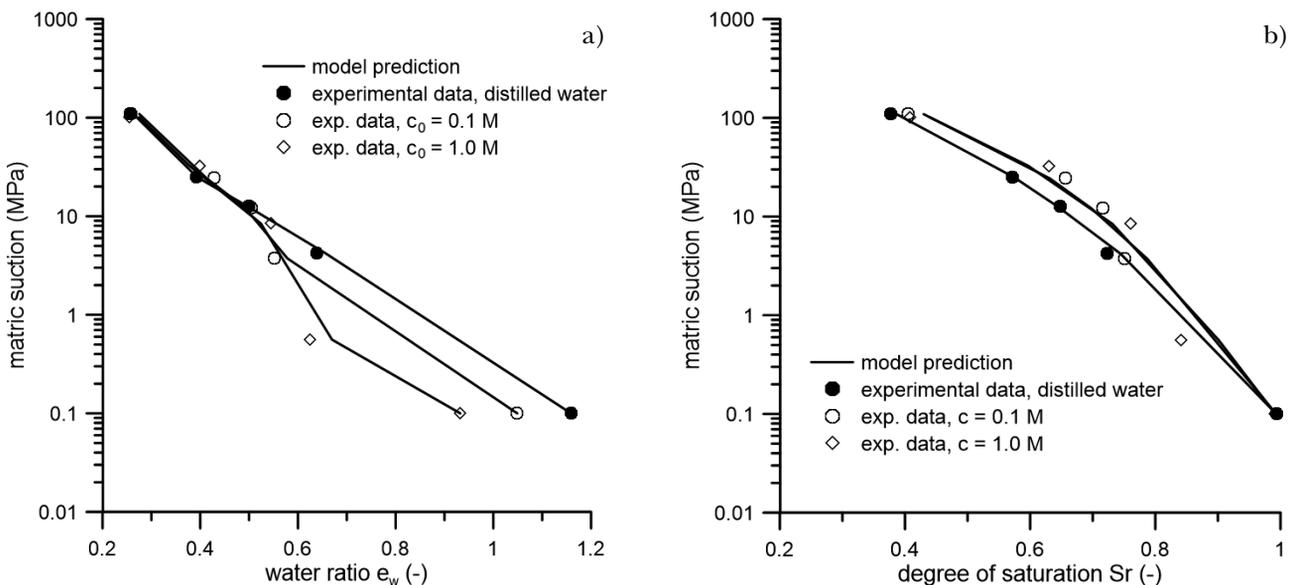


Fig. 13 – Model prediction and experimental data for specimens of GMZ bentonite dried from saturated conditions. a) water ratio evolution. b) degree of saturation evolution. Experimental data from [HE *et al.*, 2016].

Fig. 13 – Previsioni del modello e dati sperimentali per provini di bentonite GMZ essiccati a partire da condizioni sature. a) evoluzione del water ratio. b) evoluzione del grado di saturazione. Dati sperimentali in [HE *et al.*, 2016].

the kinematic constraints imposed to the porous medium upon wetting or drying: under constant volume conditions, the macro-porosity change is equal and opposite to the micro-porosity one, while unconstrained conditions imply different evolution patterns. Anyway, for a given water content and dry density, a higher water salinity – or osmotic suction – corresponds to a higher inter-aggregate void ratio. As a consequence of the evidence that the air entry value of a porous medium decreases with the amount and the size of larger pores, it follows that the matric suction associated to a given water content decreases whenever the salinity increases. Therefore, at a given density, the capacity to retain water of active clays is generally smaller when they are exposed to salty water with respect to when they are exposed to distilled water.

For modelling purposes, it was here assumed that the effects of water content and water salinity on the intra-aggregate void ratio can be superimposed and calibrated separately. Constitutive laws describing the evolution of the aggregate size have been introduced reinterpreting literature data that reported experimental evidence at the micro-structural level. Water retention tests were modelled taking into account the type of water used during the tests, as well as the evolution of the average salt concentration within the pore water. For wetting processes, this might induce non-monotonous histories of the intra-aggregate void ratio evolution, *e.g.* dominated first by the retraction due to the penetration of a high salinity water and then by the expansion due to the increase of water content. Altogether, the proposed model was found to reproduce very well water retention tests on compacted active clays as available in the literature.

Both literature data and model show that the pore fluid concentration has a strong influence on the retention behaviour of compacted active clays. At the same water ratio, the model predicts that the matric suction of a sample exposed to distilled water can be as much as two orders of magnitude higher than the one of a sample exposed to a concentrated solution. As a counter fact, at a given matric suction and initial dry density, the water retained by samples inundated with distilled water can be more than twice with respect to the water retained by samples saturated with a concentrated solution. Such strong effects are expected to have an important impact on the behaviour of compacted clay liners. As for engineering aspects, when an accurate prediction of the liner behaviour is of concern (as *e.g.* in the case of the disposal of nuclear waste or other hazardous material of high toxicity), the present model might be coupled with a transport model predicting the changes in pore fluid composition to provide reli-

able simulations of pollutant migration and of liner performance.

Acknowledgments

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Modellazione del ruolo della salinità dell'acqua interstiziale sulla ritenzione di acqua nelle argille attive compattate

Sommario

Le argille attive compattate vengono frequentemente utilizzate in molti lavori geotecnici e ambientali. Le loro particolari caratteristiche idro-meccaniche, e in particolare la bassa permeabilità, ne fanno dei materiali particolarmente adeguati per il contenimento della contaminazione dovuta a rifiuti nucleari o domestici.

Evidenze sperimentali hanno mostrato che la ritenzione d'acqua nelle argille attive è altamente dipendente anche dalla composizione chimica dell'acqua interstiziale, e che per identici valori della suzione di matrice la massa d'acqua trattenuta da una determinata argilla diminuisce con la concentrazione ionica. Questa peculiarità può avere effetti rilevanti sulla prestazione delle barriere d'argilla, dato che esse sono normalmente poste in esercizio in condizioni non sature e che la composizione chimica dell'acqua utilizzata nella loro preparazione è verosimilmente differente da quella del sito. In questa memoria vengono prima descritti gli effetti del contenuto e della salinità dell'acqua interstiziale sulla microstruttura delle argille attive, facendo riferimento a formulazioni esistenti che le trattano separatamente. Si introduce quindi una formulazione più ampia, che ingloba entrambi i contributi. Il modello che ne discende è infine validato mediante il confronto con dati di ritenzione idraulica da letteratura, che vengono riprodotti con buona approssimazione. Per le argille attive prese in considerazione, fissato il contenuto d'acqua, il modello prevede che la suzione di matrice associata a una soluzione interstiziale a elevata salinità sia fino due ordini di grandezza inferiore rispetto a quella associata ad acqua distillata. D'altro lato, per una determinata suzione di matrice e densità secca iniziale, il modello prevede che il volume d'acqua trattenuto da campioni esposti ad acqua distillata sia più del doppio di quello trattenuto da campioni esposti a una soluzione salina con elevata concentrazione.